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Burning Limits and Toughness of
Shale from the Galesburg District

Ceramics

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BURNING LIMITS AND TOUGHNESS
OF
SHALE FROM THE GALESBURG DISTRICT

BY

IDRIS NELSON

THESIS

FOR THE

DEGREE OF BACHELOR OF SCIENCE

IN

CERAMICS

COLLEGE OF LIBERAL ARTS AND SCIENCES
UNIVERSITY OF ILLINOIS

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June 1, 1915

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Idris Nelson

ENTITLED BURNING LIMITS AND TOUGHNESS OF SHALE FROM THE

GALESBURG DISTRICT

IS APPROVED BY ME AS FULFILLING THIS PART OF THE REQUIREMENTS FOR THE

DEGREE OF Bachelor of Science

in Ceramics

Ralph K. Hirsch

Instructor in Charge

APPROVED:

R S Stull

HEAD OF DEPARTMENT OF Ceramics

UNIVERSITY OF ILLINOIS

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INTRODUCTION

Purdy¹ says, "It is essential that a shale have certain physical and chemical properties in order that it may be used for the manufacture of paving brick. Of the physical properties, the more important are, plasticity, tensile strength, bonding power and fineness of grain." The first three are necessary in order that the shale may stand the treatment received in the manufacturing process. Nevertheless these properties vary greatly in degree in the different shales used for paving brick purposes. Extreme fineness of grain is not desirable, since fine grained clays have not proved to be good paving brick material. Since there is little data due to the lack of knowledge of the mineralogical constitution of clay, only one of the chemical properties, the amount of fluxing material in the shale, seems to have any definite effect upon the properties of the manufactured product. The above properties refer to the shale in the raw condition only. After burning certain other properties are essential in order that the shale may be suitable for paving brick. Brick should possess (a) toughness, (b) hardness, (c) strength, (d) homogeneity of structure, (e) freedom from lamination, (f) weather-resisting qualities, (g) regularity in form and size, and (h) uniformity in quality.

Toughness in paving brick is understood as that property of resistance to sudden stresses, as from impact without rupture. It may be considered as the opposite of brittleness.

Hardness is the ability to resist abrasion, such as the grinding action of steel tired wheels moving over a pavement.

Strength is important because the load is concentrated on a small area. The brick are frequently subjected to large transverse stresses.

¹ Purdy. Bul. 9. - Illinois Geological Survey.



Homogeneity of structure and freedom from lamination are desirable because they assure a greater uniformity of wear upon the pavement and also indicate greater toughness and strength. Otherwise the brick chip and spall badly under heavy traffic.

Weather-resisting qualities:- porous brick wear away rapidly under the freezing, thawing and wet weather conditions.

Regularity in form and shape; unless the brick have this quality, it is impossible to lay them properly .

Uniformity in quality in a given lot of pavers assures a pavement which will wear evenly over the whole surface.

In addition to these requirements of the burned product, the shale must have certain burning properties. The necessity of hardness and toughness in the brick requires burning to vitrification, understood as the point at which the brick immersed in water will not absorb more than 3% of its dry weight. Since a considerable range in temperature is found in large commercial kilns even under the best conditions of burning, it is desirable that the shale develop about the same degree of vitrification within the wide temperature limits. Thus uniformity of the product is assured.

Vitrification is due to the closing up of pore space in the burned product when it approaches the fusion stage. Each of the different mineral constituents of a clay has a different fusion temperature, and it is the softening of these various constituents when their respective fusion temperatures are approached that determines the range of vitrification for any particular clay. Experimental data has proven that this closing up of pore space takes place through different temperature ranges for different clays, also, that it depends upon the duration of the burn. Thus we find clays which have a long range of vitrification give no particular difficulty in burning but the clays which have a shorter range,

of which Illinois shales are a good example, cause considerable trouble. In either case, a temperature, beyond the maximum point of the vitrification range causes the pore space to increase again, giving an overburned product. This is known as "bleb" structure and is detrimental to the strength and toughness of the brick. To avoid the development of this "overburned" condition in all parts of the kiln it is advantageous to increase the duration of the burn and lower the finishing the temperature. The same degree of vitrification can be thus obtained as with a higher temperature in a shorter period of firing and with less danger of bleb formation. There is also danger of overburning even at lower temperatures. Brown and Murray¹ concluded that "Vesicular structure is produced by a continued burning at a low temperature as well as by excessive temperature," also, as a rule "the higher the content of fluxes, the more marked will be the influence of the time factor."

The vitrification range is determined by means of a porosity-temperature curve, the decrease in porosity being plotted against the increase in temperature. Thus clay having a long range of vitrification has a gradually sloping porosity-temperature curve while clay having a shorter range shows an abrupt drop in porosity. A technical examination of the entire deposit can be made by determining the porosity-temperature relation of samples from the various parts of the bed so that the variations of the deposit may be carefully studied and the difficulties which such variation might cause avoided in manufacture.

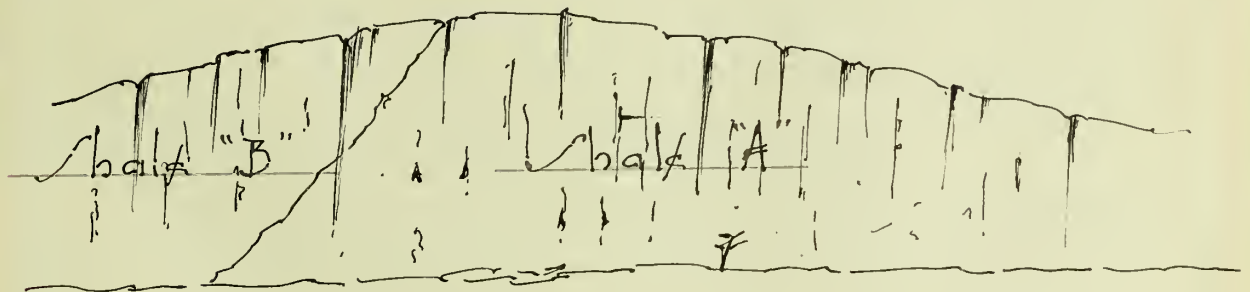
In nearly every shale deposit there is more or less variation in the working properties, in different portions of the bed due to impurities, such as thin layers of sandstone or other foreign materials and to greater weathering action in some parts than in others. With this fact in mind the writer endeavored

¹ Brown and Murray.- Function of Time in the Vitrification of Clays.
Translations: American Ceramic Society. Vol. 15, p. 193.

to establish some relation between the property of two shales taken from the same deposit and used for paving brick purposes. The two shales were obtained from the Galesburg district and will hereafter in this paper be designated as Shale A and Shale B.

Shale A is of a blue variety and belongs to the coal measure series, lying between the horizons of "coals 3 and 4." It is a deposit about fifty feet in thickness, with thin partings of soft sandstone.

Shale B is a continuation of this deposit but of a yellow nature and perhaps slightly coarser. This color is due, no doubt, to the fact that some of the ferrous iron compounds have been changed to the ferric state by oxidation and that some of the organic matter has been removed by the weathering processes that have acted upon this part of the deposit with greater rapidity than upon the remainder.



The following limits of composition of Shale A have been taken from over a hundred analyses made in the laboratory of the plant.

SiO_2	63.94 to 68.0 %
Al_2O_3	17.00 to 13.60%
Fe_2O_3	6.20 to 8.21%
CaO	.54 to 1.49%
MgO	.62 to 1.67%
$\text{Na}_2\text{O}; \text{K}_2\text{O}$	3.37 to 6.73%
Loss on Ignition	3.58 to 5.21%

From several analyses of yellow Shale B the following limits were obtained.

SiO ₂	68.59 to 72.46%
Al ₂ O ₃	13.74 to 10.67%
Fe ₂ O ₃	7.19 to 8.45%
CaO	.56 to 1.65%
MgO	.68 to 1.47%
Na ₂ O; K ₂ O	4.65 to 6.43%
Loss on Ignition	3.56 to 4.46%

From the comparison of the chemical analyses, it is found that the only noticeable differences that occur, seem to be in the SiO₂; Al₂O₃ and volatile matter. Shale B shows a higher percentage of SiO₂ and a lower percentage of Al₂O₃ and volatile matter. The loss of volatile matter can be accounted for, by weathering action and the higher SiO₂ content is probably due to the fact that SiO₂ was left when the thin sandstone layers were weathered.

Shale A is more dense and finer grained than Shale B. The disks of A average about 3.5% more in weight after being burned than those made of Shale B although the drying and burning shrinkage is the same. The approximate specific gravity of Shale A is 2.66 and of Shale B is 2.58. As to their working properties, there is no noticeable difference between them. Both have moderate plasticity and form a compact body of excellent quality in the course of manufacture.

The present study is discussed under the following headings:

- (1) Character, grinding, molding, drying and burning of test pieces.
- (2) Absorption test.
- (3) Description of Rattler test.
- (4) Conclusions.

Preparation of Test Pieces

The two samples of clay were given, as nearly as possible, the same treatment in all the experimental work. Both were ground in the dry pan and passed through an eight mesh screen. They were then tempered in a wet pan to a stiff mud consistency, passed through a small experimental auger brick machine having a round die three and one-half inches in diameter. A baffle plate was placed just back of the die to decrease die laminization as much as possible. The clay was cut into disks approximately two and one-half inches thick. These were carefully dried in a steam dryer for about thirty-six hours. Several of the disks made of Shale B split apart on drying due to the fact that the clay did not weld completely after passing the baffle plate. Both clays possess excellent drying qualities and neither showed any drying defects other than noted above.

The disks of both clays were placed in a round down draft oil fired kiln and burned together. The duration of the burning was approximately thirty-six hours, exclusive of the water smoking and oxidation periods. The temperature was raised approximately 35° per hour during the entire burn. A pyrometer was used to regulate the rate of heating and cones to determine the degree of heat treatment. The heat distribution in the kiln was kept as uniform as possible. Trial pieces were drawn from time to time and broken to determine the condition of hardness. It was noted that Shale B was less viscous than Shale A at the finishing temperature because some of the disks were kiln marked. The kiln was closed when Cone 1 was slightly bent and allowed to cool in approximately forty-eight hours.

Absorption Test

After being taken from the kiln, each disk was weighed and immersed in water for forty-eight hours, after which it was again weighed and the percentage absorption calculated. The disks were then thoroughly dried and sorted into

groups according to the percentage absorption.

Rattler Test vs. Absorption

Relative toughness of the disks showing varying percentages of absorption was determined by an abrasion test in a small experimental rattler. The rattler was a cylindrical iron mill thirty inches in diameter and fifteen inches in width, outside measurements. It was rotated at a rate of 45 R. P. M. for thirty minutes in these tests. The charge consisted of twelve disks and three hundred pounds of wrought iron balls one and seven-eighths inches in diameter. Previous experimental work with this rattler on other clays gave results which compared favorably with a standard rattler test made on the same clay under commercial conditions.

Clay A							
Test No.	Disks Nos.	Percentage Absorption	Average	Weight before	Weight in grams after Rattling	Loss	Percentage loss
I	140	.22	.227	5240	4255	985	18.78
	143	.25					
	144	.22					
	145	.22					
	147	.24					
	150	.22					
	151	.22					
	153	.22					
	167	.25					
	169	.22					
	172	.22					
	175	.22					
II	87	.22	.251	5332	4222	1110	20.8
	89	.66					
	93	.22					
	94	.22					
	115	.23					
	116	.22					
	118	.22					
	119	.23					
	129	.22					
	130	.22					
	133	.22					
	134	.24					
III	45	.50	.325	5330	4160	1170	21.95
	63	.47					
	64	.22					
	74	.22					
	109	.22					
	110	.44					
	112	.45					
	176	.23					
	178	.22					
	184	.22					
	192	.50					
	193	.22					

Clay A

Test No.	Disks Nos.	Percentage Absorption	Average	Weight in grams before , after Rattling		Loss	Percentage loss
IV	4	.45	.41	5260	4150	1110	20.11
	11	.45					
	16	.45					
	17	.45					
	22	.22					
	23	.42					
	24	.22					
	25	.47					
	26	.45					
	31	.46					
	34	.49					
	36	.46					
V	86	.46	.451	5332	4215	1117	20.95
	87	.45					
	88	.45					
	91	.46					
	96	.45					
	97	.45					
	122	.45					
	126	.46					
	141	.45					
	163	.45					
	164	.45					
	165	.45					
VI	166	.45	.59	5340	4165	1175	22.0
	170	.45					
	173	.46					
	187	.44					
	171	.45					
	174	.67					
	177	.67					
	182	.67					
	183	.68					
	185	.67					
	189	.67					
	191	.68					

Clay A

Test No.	Disks Nos.	Percentage Absorption	Average	Weight in Grams before after Rattling		Loss	Percentage loss
VII	40	.46	.64	5285	4215	1070	20.24
	50	.47					
	65	.49					
	66	.49					
	75	.50					
	77	.49					
	82	.49					
	84	.49					
	89a	.88					
	146	1.36					
	155	.92					
	168	.70					
VIII	90	.66	.67	5324	4025	1299	24.38
	92	.67					
	98	.67					
	99	.67					
	103	.67					
	104	.66					
	114	.65					
	123	.67					
	139	.66					
	148	.68					
	149	.68					
	158	.69					
IX	2	.66	.682	5305	4210	1095	20.64
	3	.69					
	5	.65					
	18	.66					
	29	.67					
	32	.70					
	51	.70					
	56	.71					
	57	.67					
	67	.69					
	68	.71					
	72	.67					

Clay A

Test No.	Disks Nos.	Percentage Absorption	Average	Weight in Grams before after Rattling		Loss	Percentage loss
X	61	.88	.864	5340	4095	1245	23.30
	62	.87					
	69	.88					
	70	.89					
	73	.86					
	83	.88					
	100	.90					
	111	.67					
	120	.90					
	125	.88					
	138	.88					
	152	.92					
XI	6	.88	.893	5370	4035	1335	24.86
	7	.90					
	13	.87					
	14	.90					
	20	.90					
	27	.90					
	28	.90					
	30	.88					
	37	.88					
	41	.89					
	47	.89					
	59	.86					
XII	8	1.10	1.22	5350	4065	1285	23.83
	9	1.10					
	19	1.10					
	33	1.38					
	39	1.13					
	46	1.13					
	52	1.39					
	60	1.37					
	71	1.36					
	78	1.09					
	79	1.37					
	105	1.09					

Clay A

Test No.	Disks Nos.	Percentage Absorption	Average	Weight in Grams before after Rattling		Loss	Percentage loss
XIII	80	1.64	1.34	5340	3935	1405	26.31
	106	1.38					
	107	1.76					
	108	1.04					
	157	1.35					
	161	1.36					
	162	1.34					
	180	1.54					
	181	1.60					
	186	1.14					
	188	1.14					
	190	1.14					
XIV	95	1.74	1.51	5410	4120	1290	23.84
	102	1.77					
	113	1.32					
	117	1.98					
	121	1.34					
	124	1.12					
	128	1.12					
	131	1.58					
	132	1.24					
	136	1.58					
	137	1.76					
	142	1.28					
XV	1	2.05	1.85	5355	3955	1400	26.13
	10	1.98					
	21	1.76					
	35	1.68					
	38	1.98					
	44	1.71					
	48	1.76					
	49	1.76					
	54	2.50					
	55	1.62					
	58	1.68					
	76	1.74					

Clay A

Test No.	Disks Nos.	Percentage Absorption	Average	Weight in Grams before after Rattling		Loss	Percentage loss
XVI	12	2.25	2.41	5305	3890	1415	26.67
	42	2.00					
	43	2.69					
	81	2.00					
	101	2.49					
	127	2.51					
	135	2.73					
	154	2.47					
	156	2.52					
	159	2.03					
	160	2.05					
	179	2.26					

Clay B

I	6	.46	.41	5075	4195	880	17.33
	7	.45					
	8	.69					
	14	.44					
	15	.25					
	16	.23					
	18	.45					
	40	.46					
	44	.48					
	45	.47					
	48	.30					
	--	.40					
II	11	.70	.885	5116	4080	1036	20.27
	12	.70					
	13	.70					
	17	.70					
	21	.95					
	22	.93					
	27	.95					
	31	.69					
	37	.95					
	41	.96					
	47	.72					
	49	.70					

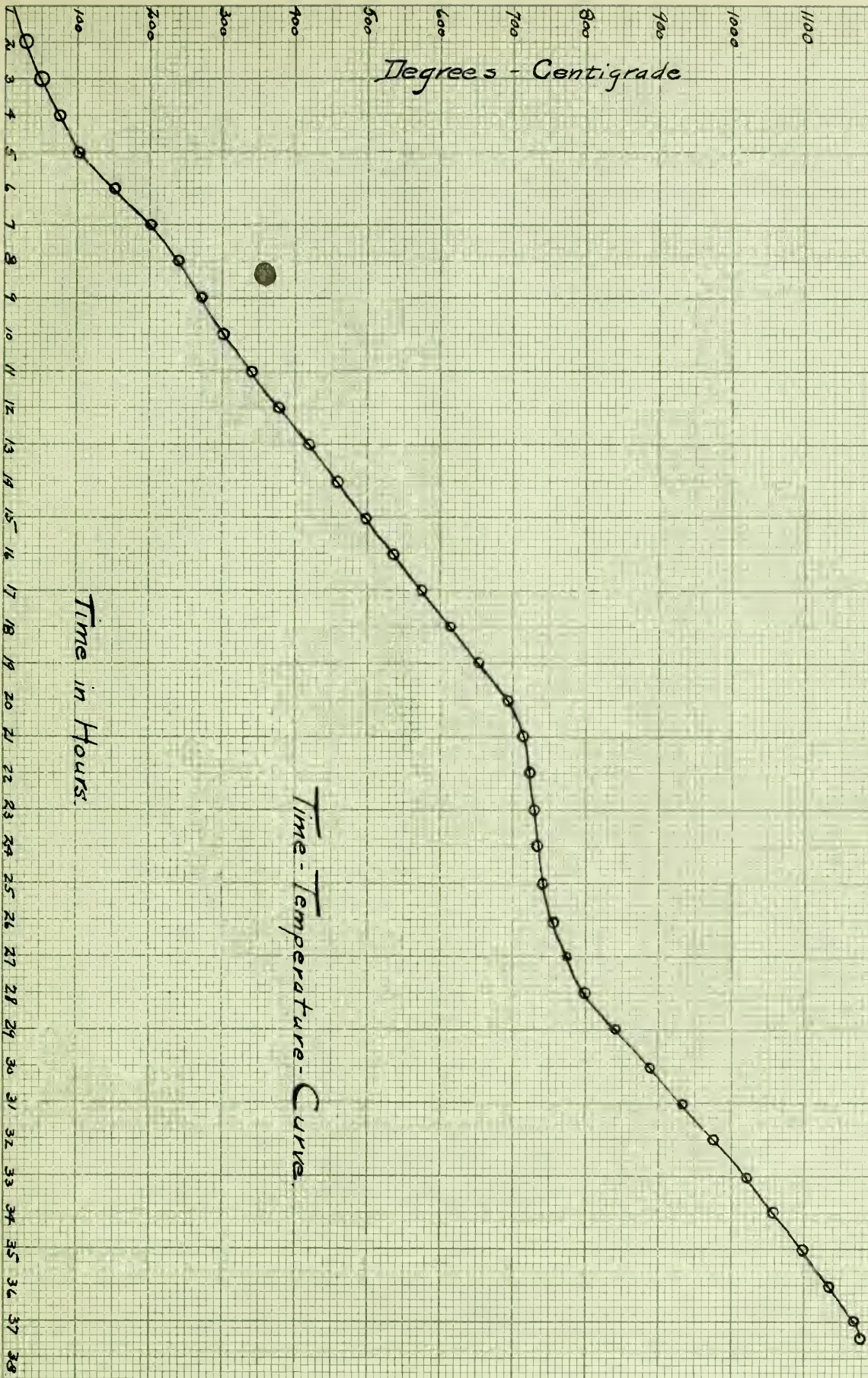
Clay B

Test No.	Disks Nos.	Percentage Absorption	Average	Weight in Grams before after Rattling		Loss	Percentage loss
III	55	1.50	1.59	5180	4095	1085	20.92
	59	1.50					
	61	1.62					
	64	1.78					
	65	1.29					
	66	1.88					
IV	1	1.64	1.93	5140	4000	1140	22.17
	9	1.66					
	10	2.08					
	19	2.36					
	20	1.39					
	24	1.16					
	25	1.74					
	26	2.60					
	35	2.60					
	38	1.81					
	42	1.40					
	43	2.56					
V	53	2.94	2.71	5155	3940	1215	23.57
	51	2.85					
	60	2.50					
	62	2.70					
	63	2.55					
	67	2.71					
VI	2	5.10	4.71	5100	3830	1270	24.90
	3	4.20					
	4	4.20					
	5	6.60					
	23	4.38					
	28	3.52					
	29	6.31					
	30	5.20					
	32	3.80					
	33	3.80					
	39	4.70					
	46	4.75					

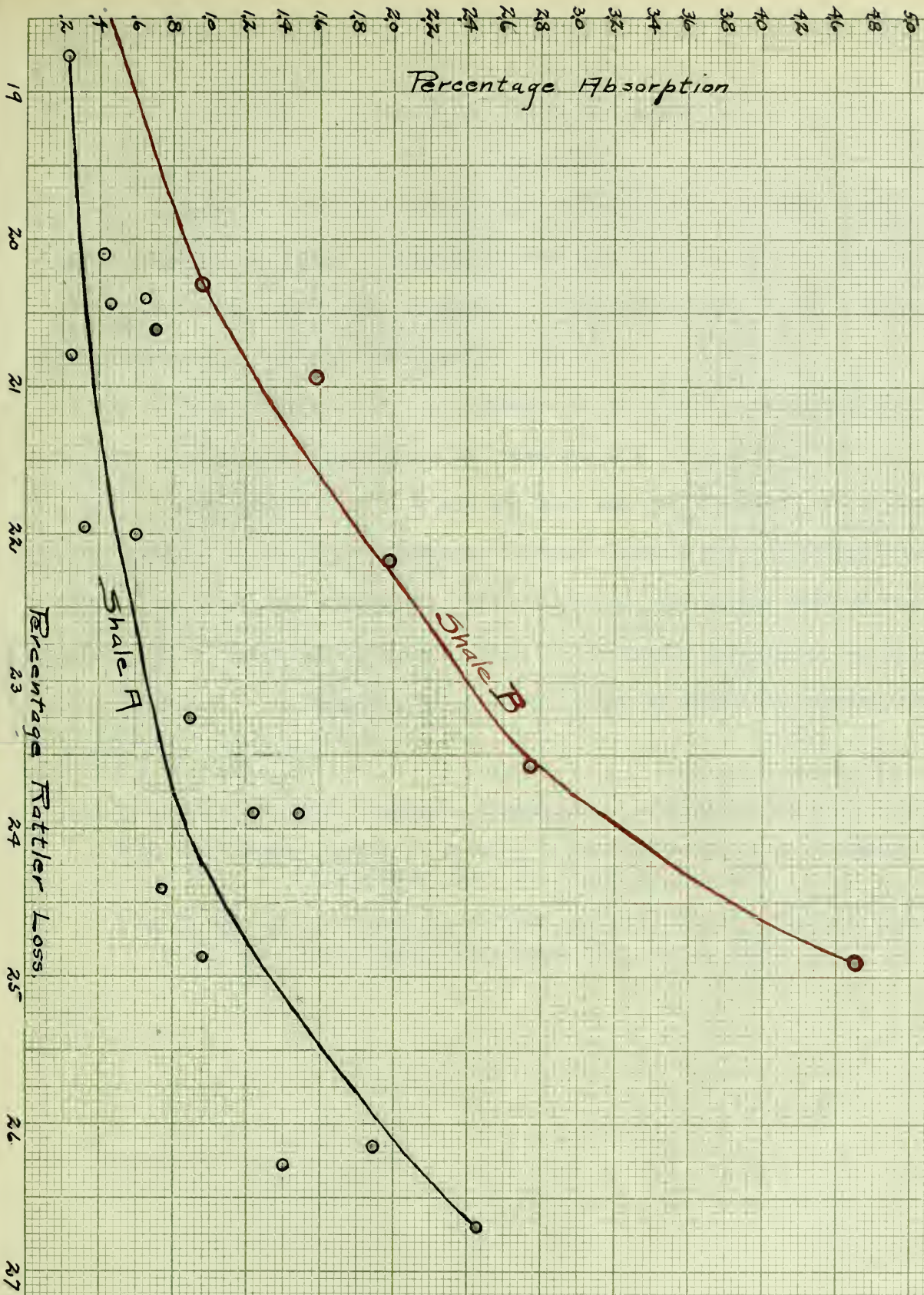
Degrees - Centigrade

Time in Hours.

Time-Temperature-Curve.







Conclusions

From the results obtained Shale B was found to be more susceptible to kiln markings than Shale A which indicates that it has a greater change in viscosity for small temperature increases. From similar data Brown¹ concludes that this indicates a shorter vitrification range.

On examination the disk appeared to be glassy, indicating that the maximum safe burning temperature had been reached for Shale B.

Although the tests do not all bear out the statement, it is probably safe to say that there is a definite relation between the percentage absorption and the rattler loss. The higher the absorption, the higher will be the rattler loss. This is especially marked after 1% absorption was passed for Shale A and 8% for Shale B. A similar conclusion was drawn by Brown² in testing clays for paving brick purposes.

¹ G. H. Brown.-A Method of Testing Clays for Paving Brick Purposes.
Trans. Amer. Cer. Soc. Vol. 16, p. 571.

² G. H. Brown.-The Viscosity of Some Shales at Furnace Temperatures.
Trans. Amer. Cer. Soc. Vol. 12, p. 265.



Disk A and B before rattling



Group of Disk A after rattling

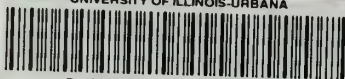


Group of Disk B after rattling





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